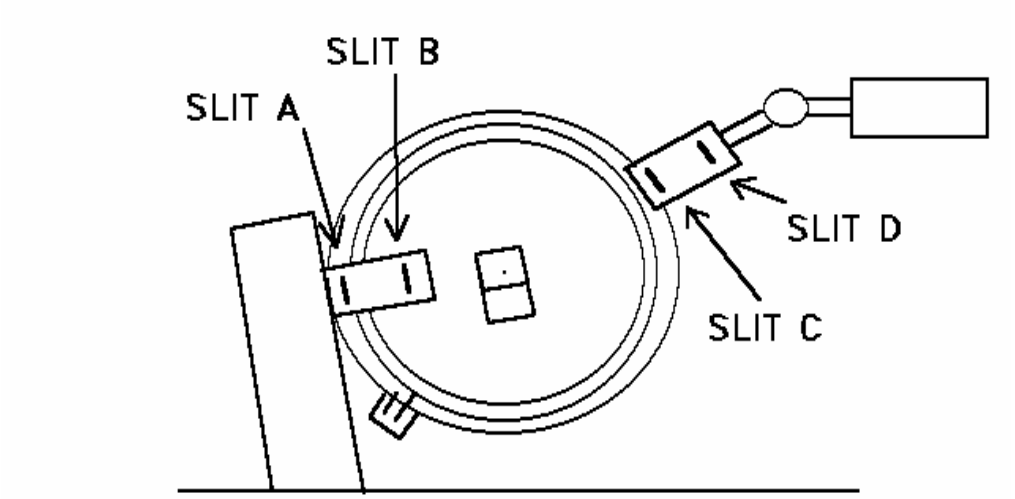
Scintag XRD Powder Diffractometer Procedures

Loading your Sample and Diffractometer Setup

Dosimeters: When working in the laboratory, always wear your dosimeter "badge" and ring(s). The badge should be clipped to clothing approximately at chest level, and the ring work on the left hand (since this will be closest to the x-ray generator). ***Only Authorized Users who have been cleared through UNM Radiation Safety Office and are wearing their assigned dosimeters are permitted to be in the lab while X-rays are being generated.***

Samples may be loaded in a variety of sample holders depending on individual needs. These may include rectangular glass slides for slurry mounts (typically used for oriented clay mounts), plexiglass side-pack or top-pack mounts, zero-background quartz plates, or various user designed mounts for thin-film or other specialized samples. Ideal sample holders are 3 cm x 3 cm plates between 5 and 10 mm thick, but the spring mounted holder allows for considerable variation. The main criterion for collection of accurate data is that the top surface of your sample be at the exact same level as the top edges of the holder.

There are several user-changeable collimating slits (see diagram below with generator at left, detector at right), and it is important that users are aware of these and insert the slits which are appropriate to their samples and the type of data they are collecting. The default slits for most routine powder data for rock samples are as follows: Slit A: 2mm; Slit B: 4mm; Slit C: 1mm; Slit D: 0.3mm.



SCHEMATIC OF FRONT OF GONIOMETER
WITH COLLIMATING SLIT LOCATIONS SHOWN

When your sample is loaded, make sure the door is fully closed.

Power-up and Power-down of Generator

Users must check to see that the XRD generator is powered-up before collecting data, and at the end of the day must power-down the generator. The purpose of this is to prolong tube life (since turning power on and off can shorten tube life). Please follow the procedure listed below:

1. When the first user of the day enters the lab, the power supply should be turned ***on*** and the Accelerating Voltage (***Kilovolts***) and Current (***Milliamperes***, upper left control) should be set to their idle values, ***-40 KV*** and ***10 MA***, respectively.
2. If the power supply is turned off (it shouldn't be), contact the Lab Manager (Jim Connolly) before doing anything.
3. Gradually adjust the MA (the top left knob) to ***35 MA*** over a period of a minute or so. Accelerating potential is permanently set at -40 KV. ***Do not adjust any other knobs on the power supply*** – there should be tape over all of the other knobs to guide you to the correct one to adjust. If the KV meter does not read -40, contact the Lab Manager immediately.
4. When done for the day check the online lab calendar to see if someone is coming in after you are done. If you are the last user for the day reduce the Milliamperes settings to the idle value of 10 MA gradually over a period of a minute or so.

Logging on and off off the EPS Network

All users of the X-Ray Diffraction system must have an account on the EPS Department Network. Both computer systems in this lab run Microsoft Windows 2000. If you do not have an account, see the Lab Manager who will give you the appropriate paperwork to complete and create an account for you.

To log on for data collection, press Ctrl-Alt-Del on the data collection computer (the one on the left), enter your Username and Password in the appropriate boxes, then click on OK with the mouse to complete the login. Data collection (using DataScan) must be done on this system. Data analysis (using Jade software) may be done on this system or (preferably) the newer faster (dark gray) system to the right.

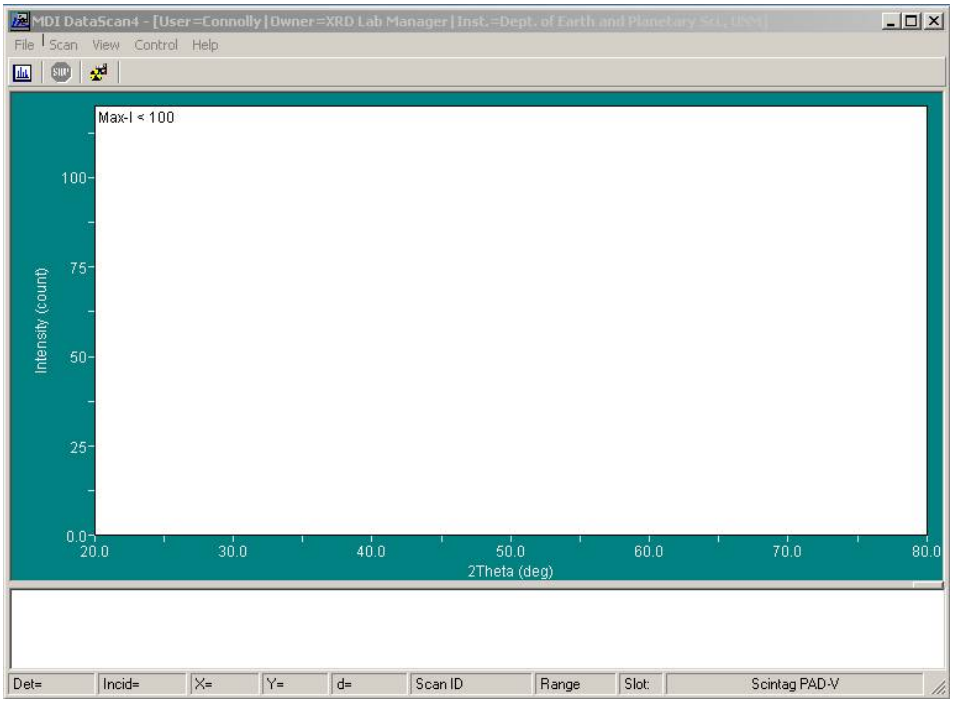
When done working and analyzing data, log off the whatever systems you are using by choosing ***Start*** Menu - ***Shutdown***, then make choosing "Log off . . ." from the drop-down list and clicking on ***OK***. (***DO NOT*** select "shut down" or "shutdown and restart" as an option.)

Data Collection with DataScan System

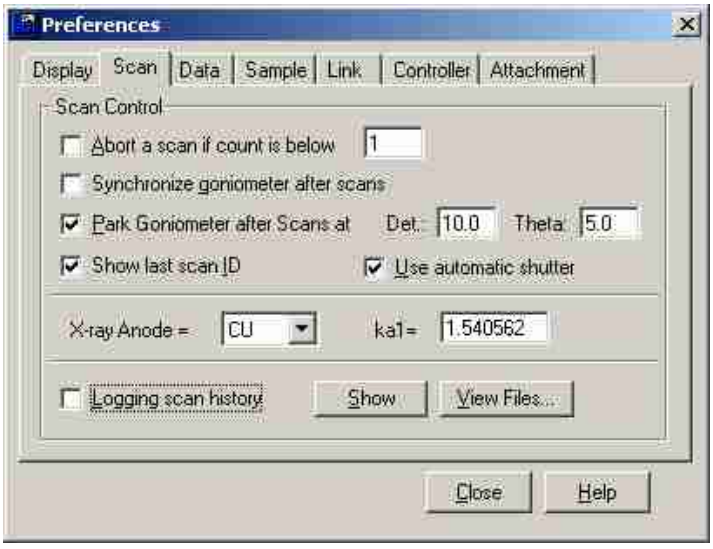
How to Set Individual Configuration Parameters in Datascan 4

NOTE: ***Each user needs to do the items in this section only once.***

In earlier versions of DataScan the default parameters for things like angular limits and whether the system "parks" at the end of analyses were set the same for all users. ***In DataScan4 user must set their individual preferences for how the diffractometer operates.*** This only needs to be done one time by each user of the system, but it must be done to insure proper operation of the system. Follow the procedures below to insure that the goniometer will be parked at the 10° 2θ exchange position when the analysis is done and to set the upper and lower angular limits.

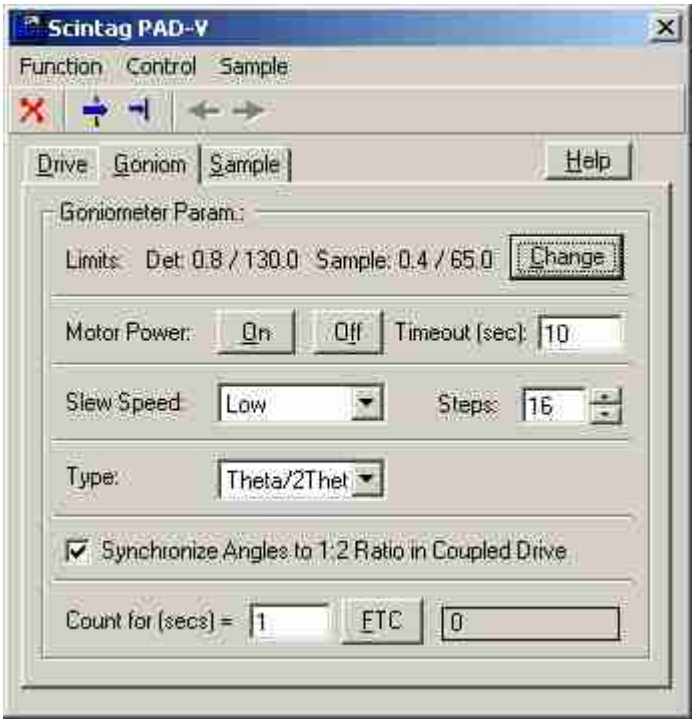


Start DataScan4 from the "MDI DataScan4" icon on the desktop. On the startup screen (at left), select "Control" on the Menu, then "Preferences." In this window, click the "Scan" tab to bring up the window below.



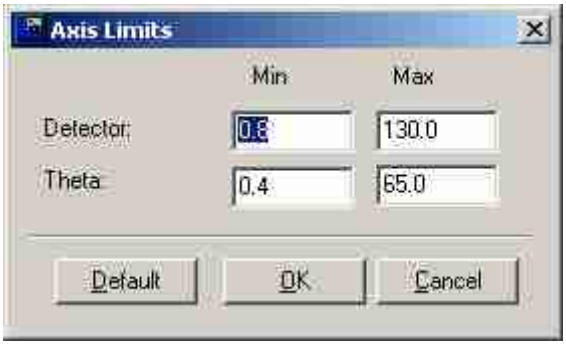
In the window at left, ***check*** the box that says "Park Goniometer after Scans at" and enter 10.0 in the "Detector" box and 5.0 in the "Theta" box.

When this has been properly set, close the "Preferences" Window to return to the main DataScan Window.



Again select the "Control" menu in the main DataScan Window and select "Databox" to bring up the "Scintag PAD-V" window shown at left.

The default detection limits will generally be set with 2° as the lower limit and 160° as the upper limit. To enable scans below 1°, click the "Change" button to bring up the screen below.

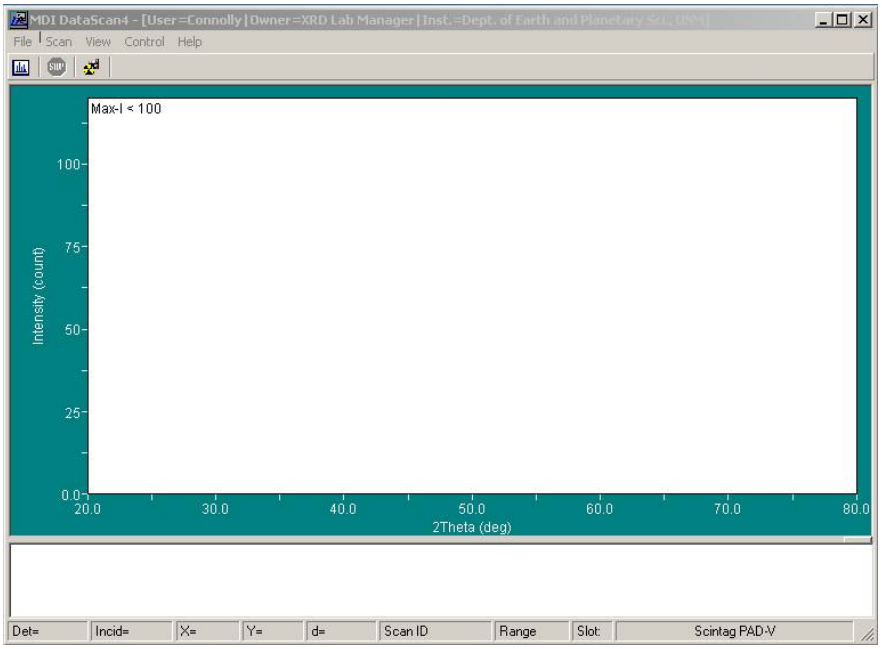


Enter the values shown at left in the boxes:
Detector: Min: 0.8; Max: 130.0
Theta: Min: 0.4; Max: 65.0

The lower limits given are the absolute minimums; setting limits lower that this can cause damage to the detector. The upper limits can be set somewhat higher (160° 2θ or "Detector", 80 ° θ) if required for specialized work.

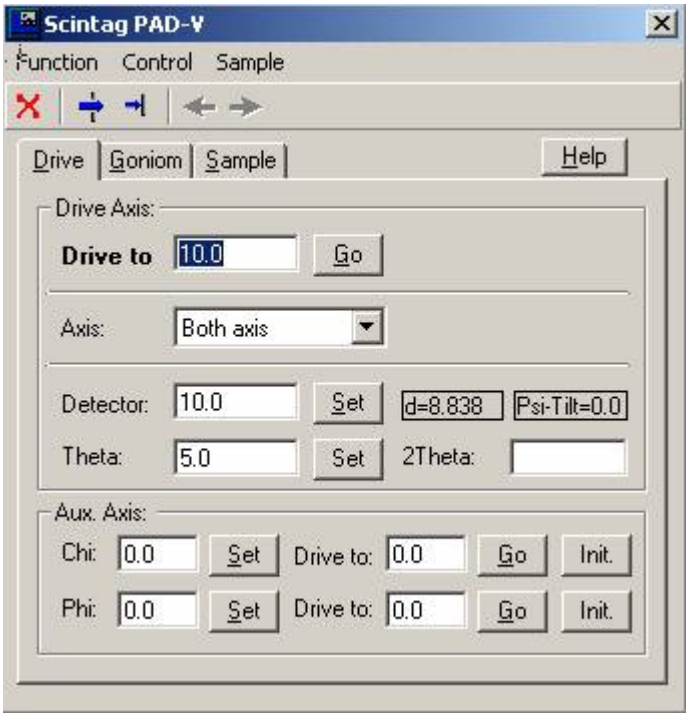
Click OK to make the changes permanent. Remember, these settings only need to be changed one time for each user.

Startup and Calibration Check



After you have logged into the data collection system, double-click on the "MDI DataScan4" icon on the desktop. After a few seconds you will see a screen that looks very much like the one to the left.

The next step is to make sure that DataScan and the goniometer agree on the 2θ and θ positions. From the DataScan menu, choose "Control" and "Scintag PAD-V". The Window shown below should appear. Make sure the tab named "Drive" is selected.



Note the positions shown in the middle of the window for 2θ (Detector) and θ (Theta). Normally these will be 10.0 and 5.0, respectively. ***Walk over to the diffractometer*** and make sure that the positions shown are identical to those shown on the goniometer. As you face the goniometer, the inner ring reads the θ position, and the outer ring reads the 2θ position. The micrometer scales on the top of the goniometer show each degree divided into 100 divisions, the left for 2θ and the right for θ. These should both read zero (within ±3/10 of a division). ***If these conditions are met, close the window and skip to the section, Data Collection.***

You may enter a 2θ value in the "Drive to" box to manually drive the goniometer to any desired location within the angular limits between 1º and 130º 2θ. The blue arrows at the top of the screen may used to open (arrow through "window") or close the shutter. ***All***

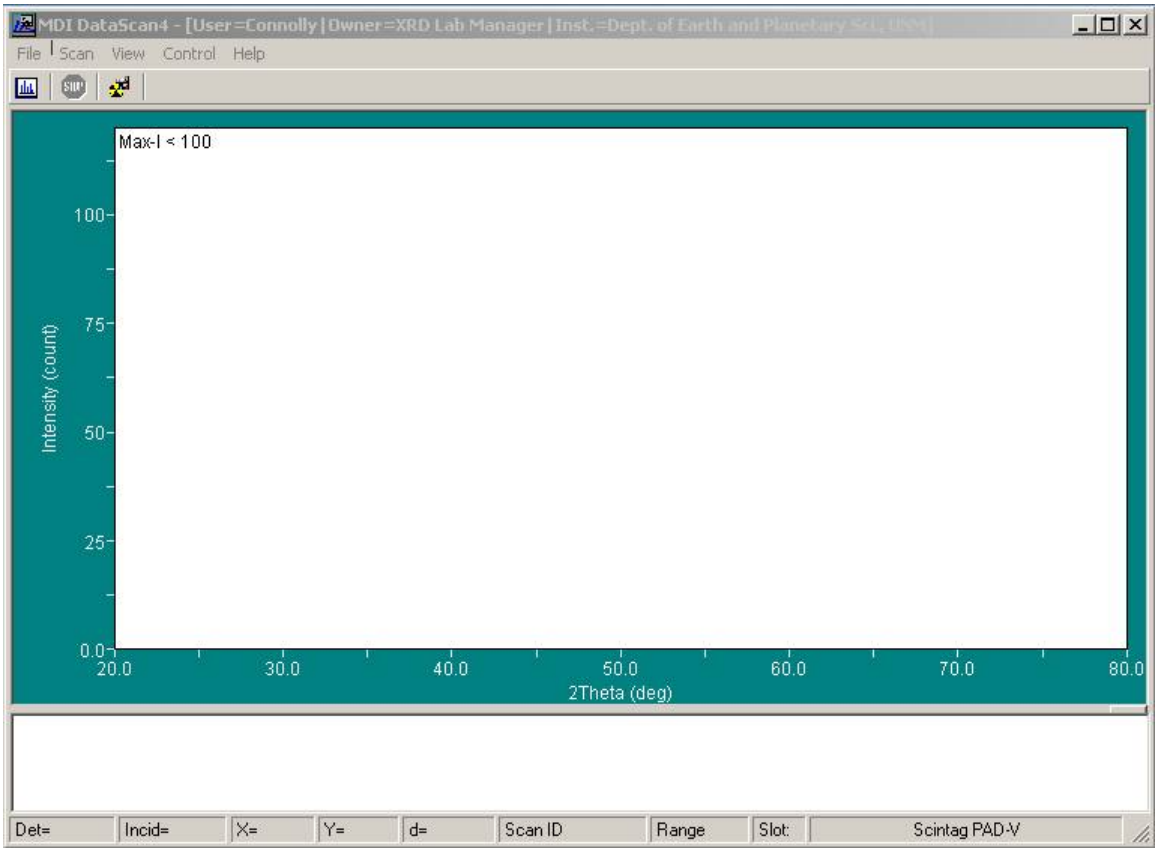
other settings in the controller window are for lab administration use only and should never be changed by users.

If the positions do not agree, you have two choices. You may contact the lab administrator (Jim Connolly, Connolly@unm.edu, Office Northrop 209A) who will recalibrate the angular positions or you may use the following calibration procedure:

1. In the controller window, from the "Control" menu select "Init. Goniometer". The program will ask you to confirm that you want to calibrate the angle positions. A "Calibrate Angle" command will then be sent to the goniometer. This will cause the θ and 2θ to be (independently) driven to the next whole-angle position.
2. When the goniometer has done moving, a dialog will pop up asking you to enter the angular position. Reading the large dials and the micrometer scales (using the black scales not the red ones), enter the positions to three decimal places (i.e., 10.997)[1]. This is done for θ and 2θ independently. When you make these entries, the step-gearing correction will usually modify your entry slightly, so don't be alarmed.
3. After this is done, enter a few angles in the "Drive to" box and check that the goniometer drives to the entered 2θ and θ angles accurately. If it is off significantly, repeat these steps until your angles stay within ±.003 deg on both θ and 2θ. ***If you cannot achieve this, stop what you are doing and contact the lab manager.***
4. To check shutter function, click on Open Shutter (the big blue arrow) and check that the shutter opens (i.e., X-Ray on light comes on). Click on Close Shutter (smaller blue arrow) to close the shutter (X-Ray light goes off).
5. When done, close the Window (the red X or Function – Close on the Menu) and proceed with data collection.

[1] The DataScan 4 calibration routine has a glitch in how it calculates θ. The command sent is to move each axis independently to the next whole angle, but the software always assumes that θ is half of 2θ and will always present the wrong number in the calibration routine. Carefully observe what the correct angle is and always enter the correct number for θ and all should be okay. Make sure to enter several 2θ values into the "Drive to" box and make sure the system knows where it's at before proceeding.

Data Collection Overview



The DataScan program window above has several parts, from top to bottom:

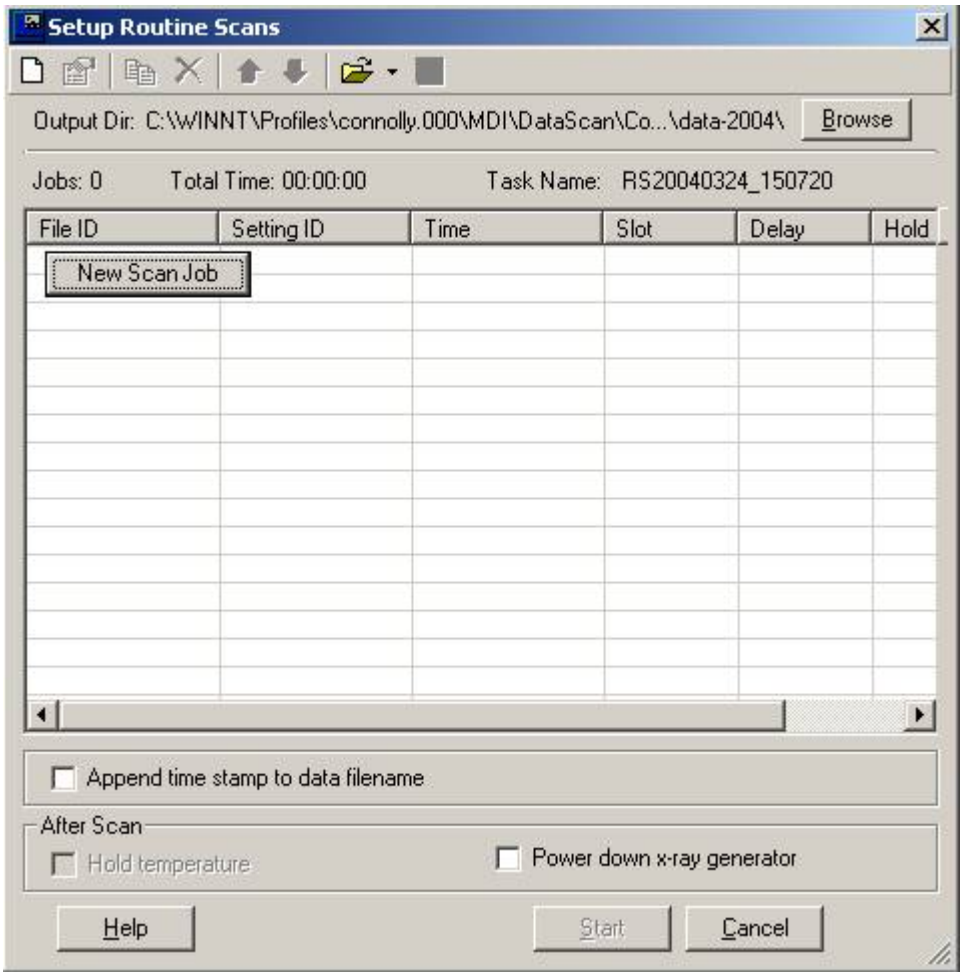
- The title line (colored, top of windows) indicates the Program Name, Current user name, and registered owner of the software.
- The Menu line below includes ***File***- and Printing-related items, ***Scan*** (setup and start scans), ***View*** (items related to how data are displayed as collected), ***Control*** (items related to direct control and configuration – primarily for lab administrator use), and ***Help*** (to access Windows Help. Note that may not be up to date for the version of the program that we have).
- The icons below provide, left to right, direct access to setup a routine scan, quick stop for a scan in progress, and direct access to the controller window.
- The display window provides a graphical display of the current data collection activity
- The message window provides a real-time log of DataScan to Diffractometer communications and activities (including errors).
- The bottom line provides real-time information about active data collection including Detector (2θ) position, Incident beam (θ) position, the mouse cursor positions X (=2θ), Y (=intensity), and d (d-spacing), the ScanID (filename) for the job, and the Range parameters for the scan. Slot is for a sample changer that we do not have.

DataScan includes 3 modes of data collection, ***Quick*** scan, ***Routine*** scan and ***Area*** scan.

- The ***Quick*** scan can be used to quickly enter run parameters in a "Scan Range" window and collect data from a specimen loaded into the diffractometer. ***At the conclusion of Quick scan data collection, the user must save the file (in .MDI format) by the user with a File-Save command or it will be lost when you quit the program.*** It is only useful for rapid reconnaissance to see if there is a peak located in a particular limited range prior to running a routine scan.
- The ***Routine*** scan creates a setup table and automatically saves data as it is collected. This is the mode that should be used by lab users. It provides more control (allowing use of saved setup routines for data collection) and lets the user enter more information about their sample into the data file.
- The ***Area*** scan is a special type of scan that is not generally used in our lab.

Routine Scan Setup

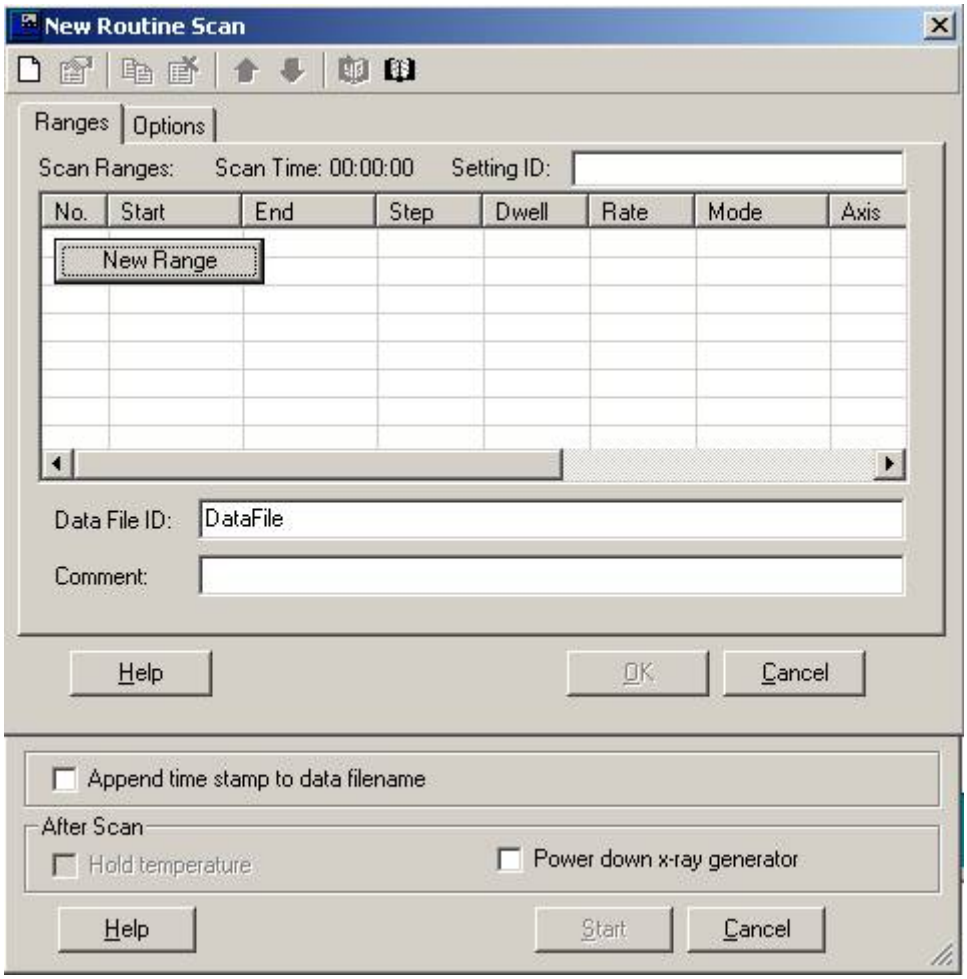
From the DataScan menu, choose "Scan" – "Routine Scan" which will bring up the scan setup window show below.



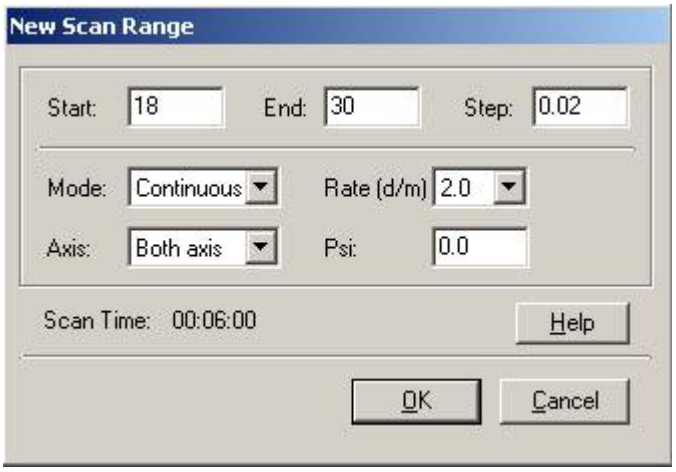
Select your Output Directory: By default, each DataScan user has a personal data folder on the local (C:) drive on the computer (with the *very long* path to that folder shown on the window above). ***The first time you use the system you should change this Directory to your "L:" drive folder.*** Click on the ***Browse*** button, and enter "L:\" in the location window and then pick a folder in which to save your data. Entering a new folder name in the "path" will cause the new folder to be created. Using the "L:" drive allows you to access your data from any system on the network, including the data analysis system to your right. Local (C: drive) data storage is not available on the network. The only time to use local data storage is if there is a scheduled network outage on a night on which you are collecting data overnight.

Important Note for Lab Users: Scan setups (i.e., how your scans are done) are customized for each user, so each user needs to recreate and save their own settings for the scans that they do routinely. What follows are the basics.

The first time you setup a routine scan, you may click on the "New Scan Job" button to define a scan. This only appears on a blank setup window. Subsequently you may use the "Page" icon in the upper left, or right-click on a blank line to setup a new routine scans, and bring up the window below:



As with the previous window, there is a "New Range" button on a blank page, or you can click on the "Page" icon to define a new scan range (or use your right mouse button to define a "New" range): The window below will appear:



On the scan range dialog, the following are defined:

- Starting angle (2θ)
- Ending Angle (2θ)
- Step Size (minimum 0.01º; 0.02º generally best for continuous scans, 0.05º for step scans)
- Data Collection Mode (Continuous, or Step Scan)
- Rate (degrees/minute for continuous mode) or Dwell (time in seconds per step in Step mode), and Axis (both axis for coupled scan).

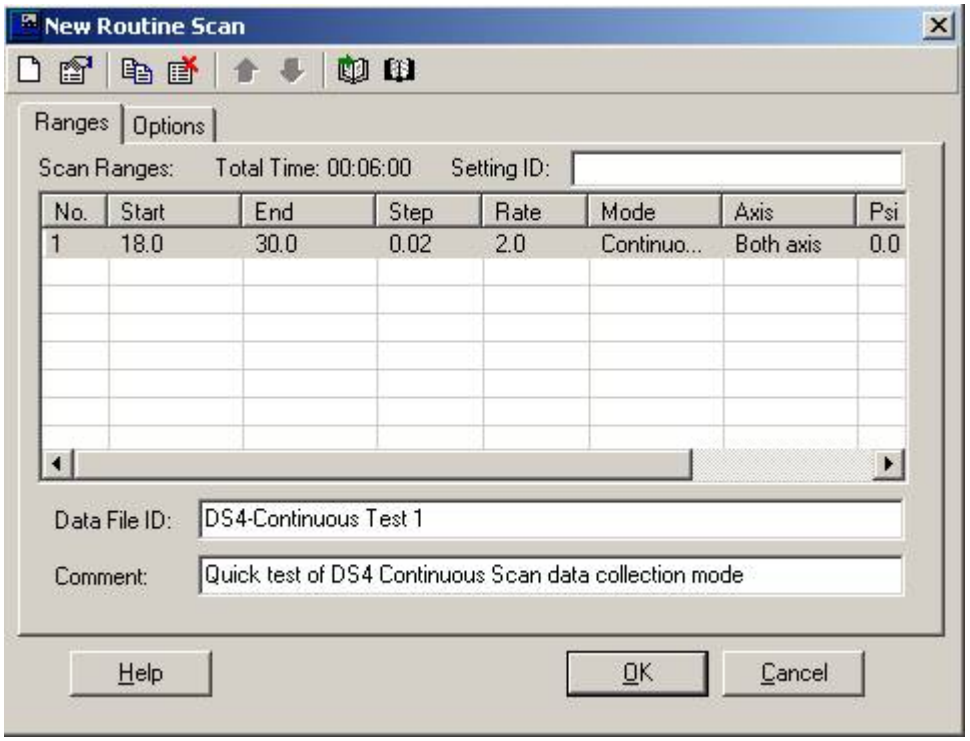
The "Scan Time" will be calculated when sufficient parameters are entered. Click OK in window to return to the New Routine Scan window (below) with collection parameters defined.

A Timing Note: For step scans, you must enter the dwell time per stop (rather than degrees per minute) as a timing parameter. For reference when entering values, a step scan with a step size of 0.02 and a dwell time of 1.2 sec/step is equivalent to a 1 deg/min continuous scan.

When to use Continuous Scan vs. Step Scan: A continuous scan collects data continuously by sampling the detector at intervals throughout the scan and as such does a kind of electronic smoothing of your data as it is collected. A step scan moves the detector in steps and counts and records data as it collected.

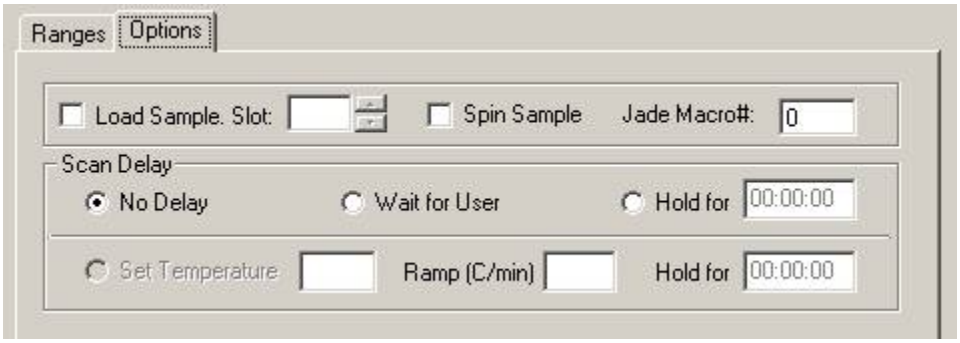
Rule #1: For scans slower than ½ deg/sec (with a 0.02 step size), always use the Step Scan mode. This will prevent calculation overload that can occur with DataScan when doing very slow step scans.

Rule #2: For scans faster than ½ deg/sec (with a 0.02 step size), always use the Continuous Scan mode. This will prevent data errors associated with the mechanical "settle down" time that occurs with stepped movements of the goniometer.

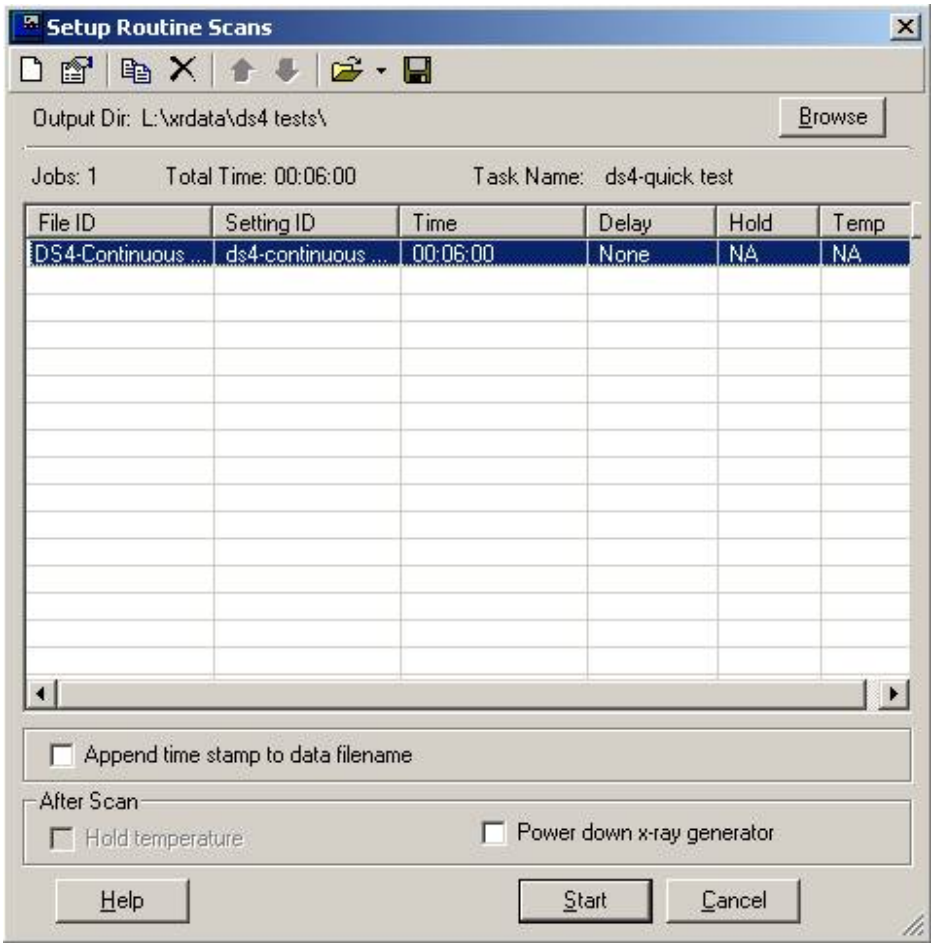


Saving Scan Parameters: The scan parameters may be saved to a separate file in your personal "Scan Book" by clicking on the save parameters icon (the open book icon with the arrow), so that the parameters may be reused for other scans. The open book (without the arrow) may be used to retrieve previously saved setups from your "Scan Book".

In the "New Routine Scan" Windows enter a Data File ID (this is the filename to be used and may be a long filename) and a Comment (this is used for your description of your specimen). The "Options" window may be used enter a "wait for user" delay should you choose to setup multiple specimen with you acting as the sample changer.



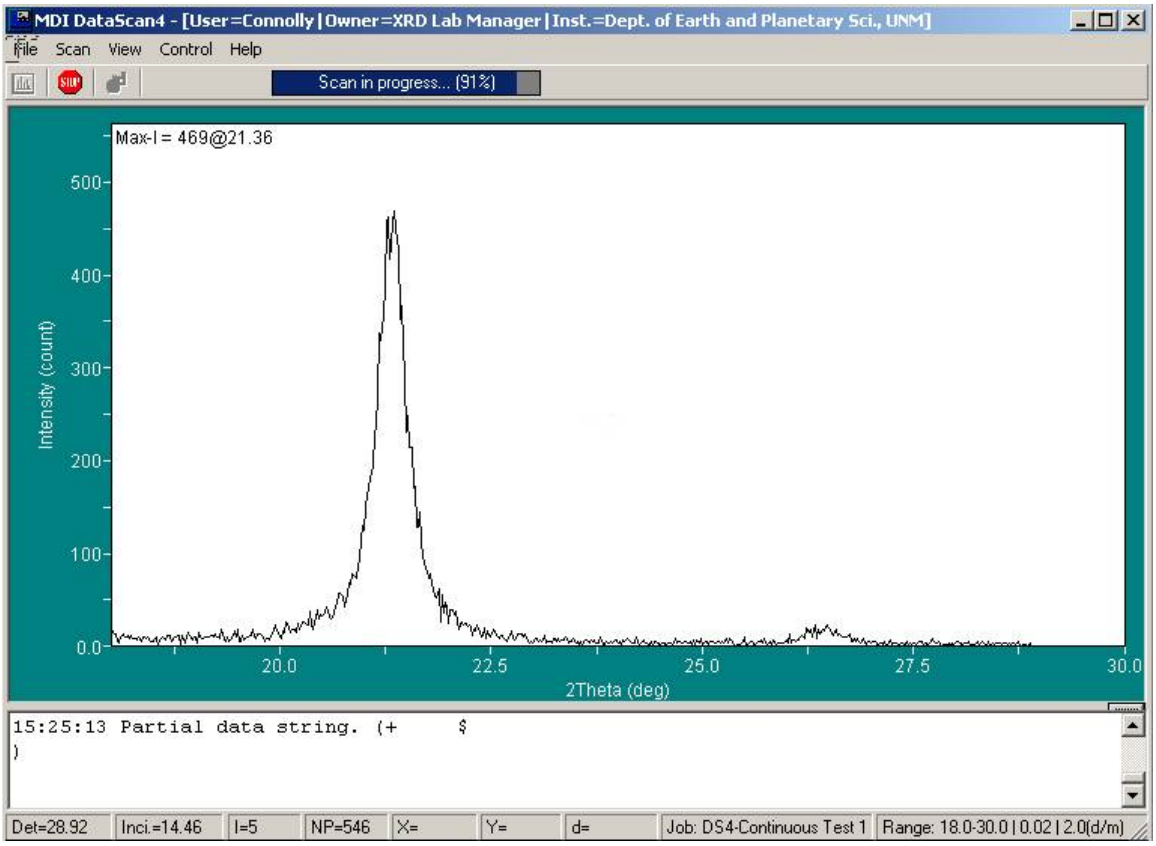
Click OK on this window to bring up the completed setup window (below):



IMPORTANT NOTE:

Only one scan range (i.e., one line) should be on this screen before starting your analysis. Multiple lines are used only for systems with sample changers – something that our system does not have. ***If you have multiple lines, remove those that are not related to the specific analysis you want to do now.*** When the system runs the jobs, each line on the screen is treated as a job and all job lines will be run in sequence.

Click on the Start button to begin data collection. The program pops up a window confirming the current goniometer position. It is a good idea to quickly check before data collection starts that this position is correct. When this window is cleared, data collection starts and the DataScan data collection window opens. In the window below, data collection is about 90% complete.



When data collection is done, the display written and the diffractometer is parked at the 10º 2θ "exchange" position*, exit the program (***File*** menu – ***Exit***). If you are finished using the system, log off the network to close your network connection.

* Diffractometer will only park if you have set this on your default settings; see page 3 for how to set your defaults properly.

DataScan 4 Capabilities

I am just learning some of the important differences between the old and new versions of Datascan. A few of these differences are noted below.

- Every user has their own settings for custom scan setups and data storage locations. These are not shared between users as in the older version of the program.
- If you save data to a network location, the next time you run the program it assumes you will save to the same location. This makes network drive use a lot easier.
- "Quick Scan" can be used to quickly scan a small angular range of interest (i.e., if you are looking to see if you have peaks in a small 2θ range) before doing a full routine scan. This can be useful to see if it is necessary to run a full "Routine" scan.
- There are now some nice capabilities in refining scans and zooming in on areas of interest. The DataScan display Window is like a mini version of a Jade zoom window and you can zoom in on an area of interest by dragging a box around it with the mouse and then "Scan this range . ." from the Scan menu to get better resolution of areas of interest. This is done as a "Quick Scan" and must be saved as a separate file (if you want to keep it).
- You may print your data directly from DataScan if desired. Make sure the printer is set up to print in "landscape" mode. Printouts are basic, but give you quick printed output of your data without requiring Jade.
- The view can be modified to show dots (at each of your "step" points) or a rectangular grid overlay.
- The software now also includes a pattern viewer (File – Pattern Viewer) that opens in a new Window and lets you view your collected data patterns. This pattern viewer appears to have some very useful data viewing capabilities. Feel free to explore what you can do with it.

A request for all users: Although we have passed our free support period for DataScan 4, we still would like to know about any quirks that you discover about the program that future updates should change or correct. Things that should be reported should include:

1. Anything that doesn't work correctly
2. Any quirky behavior of the program – i.e., loss of data communication, lost data, excessive errors in the log window, etc.
3. Any improvements or enhancements you would like to see in the program.

Please report anything to Jim Connolly in person or by Email to connolly@unm.edu.

Enter User and Scan Information in XRD Log Book

When data collection for the day is completed, fill out the logbook recording what was done. All logbook entries (at a minimum) should include

- Today's ***date***,
- Your ***full name*** (printed legibly – not your nickname, username or partial name),
- The ***number of samples run*** (only include ***successfully*** completed runs in your total, plus run type, i.e., step scan, continuous scan, etc. These are runs for which you have a data file saved),
- Notes about condition (***OK*** if all is working appropriately, an expanded note if something needs attention. If you have any failed scans which you had to redo, please note this here. Each logbook page has a block at the bottom of the page with room for detailed operational comments.),
- Your ***Supervisor*** and/or Account Number for use in billing. All users should have a PR on file for UNM work, a PO for outside work, or have made billing arrangements with the lab manager. If you do work on multiple accounts, each needs to be identified on a separate line. You ***must*** print this information ***legibly and accurately*** since it is used in billing for lab use.

Note: Not making logbook entries or making false entries are cause for immediate termination of lab privileges.

Data Analysis with Jade Software

As of this writing Jade 5 is installed on the data collection computer (with the 1999 IDCD database) and Jade 6.5 (with the ICDD 2003 database) is installed on the data analysis computer. Hopefully by January 2005 we will have Jade 6.5 on both systems with the new 2004 ICDD database.

This document is concerned with data collection, not data analysis, and users are referred to the Introduction to Powder Diffraction course page for an introduction to Jade. (See http://epswww.unm.edu/xrd/xrd-course-info.htm, Week 11.)

The paper copy of the Jade 5 manual has disappeared from the lab. We have put shortcuts to the Jade 5 "Help" file, and the Online Jade 5 manual on the desktop of the Data analysis (Gray Dell) system. These two files contain virtually everything found in the missing Jade 5 paper manual. If individuals would like a copy of either or both files for use on their own computers, see the lab manager about how this may be accomplished.

The Jade 6 help files are copy protected and can only be used on the systems actually hosting the software. The Jade 5 help files do not contain everything in the newer version, but do contain most of what users need to know about how the program works.

A large suite of advanced tools are available in Jade including profile refinement, advanced search-match and many other things which your lab manager has not yet mastered. We also have the MDI Shadow program which will do various types of profile

fitting, line-shape analysis, and crystallite size analysis. Users with specialized data analysis needs are encouraged to read the Jade 5 (online) and Shadow (paper) manuals which are found in the laboratory. If you need help getting the program to do something, please feel free to contact the lab manager. I am far from an expert with all aspects of Jade, but will help as much as I can.

Printer Tips and Tricks

The Epson Color printer to the right of the computers is now the default printer for both data collection and analysis computers. This printer is fairly quick and produces 360 dpi output on plain paper. Here are a couple of things to be aware of:

- **The printer should be off when you enter the lab. Please leave it off when you leave.** Leaving the printer on and unused for an extended period of time can cause the ink to dry up severely clogging the print heads and causing fatal damage.
- **The first page of the day takes a while to come out.** When first turned on, the printer may take a few minutes to warm up. This warmup routine is identified by a bunch of whirring and clicking during which no pages come out. ***Be patient.*** After it warms up, it does 2 to 3 pages per minute in color and about 5 ppm in black.
- **If there are streaks in your printouts,** the printer needs to go through a print cleaning cycle. Start any program from which you can print and access "Printer Setup" – Jade 5, Microsoft Word, etc. Execute a print command (File – Print in Word, or "Print Setup" in Jade 5 followed by right clicking on the Print button to pull up the setup dialog), then click on the "Properties" button to the right of the printer name, and select the "Utility" tab in the Window which pops up. To correct the problem, run the "Nozzle Check" then Head Cleaning and/or Print Head alignment as needed.
- **If the Printer Stops Printing in the Middle of a page do not attempt to physically pull the page out.** This can damage the printer. The proper course of action is to first press the "Eject" button (with a page symbol above it) to eject the page. If that doesn't work, turn the power switch on the printer off, leave it off for a few seconds, and turn it back on. The page should eject during this process.
- Paper may be found in the lab in the cabinet on the opposite side of the room. If "Ink Jet" paper is available, it may be used but regular 20lb bond works fine in the printer. Special paper to print at higher resolution (720 dpi), and photo-quality resolution (Dithered 720 x 1440 DPI) and as transparency may be purchased at the bookstore or any other source and used in the printer. At this point the lab will not be supplying these high-resolution papers to users.
- If any of the color indicator lights on the printer are flashing (typically meaning low or out-of ink condition) contact the Lab Manager, Jim Connolly.